



***In vitro* Comparative Study of Adhesion Force in Dentin of Three Cement Sealers BC-Sealer, AH-Plus and MTA Fillapex**

Ana Gabriela Carrillo-Varguez^{1*}, Tanya Bustamante-Reynoso¹, Luis Hernan Carrillo-Varguez¹, Miguel Angel Cadena-Alcantar¹, Benjamin Gonzalez-Vizcarra², Ricardo Valdez-Castro³, Luis Jesús Villarreal-Gómez^{2,4}

¹Facultad de Odontología, Universidad Autónoma de Baja California, Tijuana, México

²Escuela de Ciencias de la Ingeniería y Tecnología, Universidad Autónoma de Baja California, Valle de las Palmas, México

³Centro de Investigación y Desarrollo Tecnológico en Electroquímica S.C. -Queretaro, Queretaro

⁴Facultad de Ciencias Químicas e Ingeniería, Universidad Autónoma de Baja California, Tijuana, México

ABSTRACT

Sealants based on calcium silicate have the ability to provide excellent sealing and bioactivity. Usually, it is recommended to be used in a single cone technique. The purpose of this study is to evaluate the adhesion forces of EndoSequence BC-Sealer® - a bioceramic based premixed calcium silicate-phosphate (BC; Brasseler USA, Savannah, GA), compared with a cement-based MTA (Mineral Trioxide Aggregate) Fillapex® MTA (Angelus), and a cement based on epoxy resin AH-Plus® (DeTrey/Dentsply, Ballaigues, Switzerland). The objective of this project, is to compare adherence to dentin between filled teeth with single cone technique (CU) BC-Sealer®, lateral condensation (CL) MTA Fillapex® and AH-Plus®. For this, 45 uniradicular extracted teeth, palatal roots of upper molars and distal roots of lower molars with large and straight canals were used, they were randomly divided into 3 groups (n=15), Group 1, BC-Sealer® CU; Group 2, MTA Fillapex® CL; Group 3, AH-Plus® CL. The roots were cut into specimens of 4 mm thick in the middle and apical thirds, leaving 30 specimens per group and the adhesion strength was measured using a standardized compression test. As a result, Group 1, BC-Sealer® CU had the bond strength statistically superior to Group 2, MTA Fillapex® CL and Group 3, AH-Plus® CL. Finally, it was concluded that BC-Sealer® CU material proved to be the best adhesion in both thirds of the root canal being significantly noticeable in the middle third, compared to MTA Fillapex® CL, and AH-Plus® CL.

Key words: Sealants, adhesion forces, BC-Sealer, AH-Plus and MTA Fillapex

HOW TO CITE THIS ARTICLE: Ana Gabriela Carrillo-Varguez*, Tanya Bustamante-Reynoso, Luis Hernan Carrillo-Varguez, Miguel Angel Cadena-Alcantar, Benjamin Gonzalez-Vizcarra, Ricardo Valdez-Castro, Luis Jesús Villarreal-Gómez, In vitro comparative study of adhesion force in dentin of three cement sealers BC-Sealer, AH-Plus and MTA Fillapex, J Res Med Dent Sci, 2018, 6 (4):6-11

Corresponding author: Ana Gabriela Carrillo-Varguez

e-mail✉: anagabriela@uabc.edu.mx

Received: 01/09/2017

Accepted: 24/10/2017

INTRODUCTION

One of the keys for a successful root canal therapy is an appropriate filling procedure [1]. Historically, the sealing of the duct system has been achieved with gutta-percha and cements [2]. The purpose of the obturation is to provide a conduit filling in all dimensions in order to create a fluid apical seal to prevent entry of bacteria and their toxins in the periapical tissues [3].

Several studies have been developed to evaluate the performance of root canal filling [4-8]. The main objective of a root canal treatment is to obtain a three-dimensional hermetic seal of an apical and coronal root canal level, this is the key for a successful endodontic treatment that prevents contamination.

The use of sealant cements in combination with gutta-percha, either single cone technique or cold condensed, is fundamental to achieving the objective of root canal treatment, because it has no binding properties dentin regardless of the technique used. Eliminating the dentinal residues produced during biomechanical preparation of the root canal, it is possible to adhere the sealing material to the dentine walls, and simultaneously make the luting cement from penetrating into the tubules, creating a monoblock, that is to say, the solid core seal and sealant cement form a unit, which fills at the same time, both the root canal and dentinal tubules.

Finally, it is important to compare the currently used materials with the new ones that are coming to market to have more options with considerable advantages that may increase the rate of successful treatment (Table 1). Therefore, the purpose of this research was to evaluate the adhesion properties of a new bioceramic sealer EndoSequence® BC using BC Sealer™ Points, using condensation techniques with single cone, lateral

condensation with AH Plus® and lateral condensation with MTA Fillapex®.

METHODOLOGY

Samples

Forty-five uniradicular-extracted teeth, palatal roots of upper molars and distal roots of lower molars with large and straight canals were used, stored in chloramine T solution at room temperature. Clinical crowns of the tooth bodies were removed with a diamond disc at low speed; they were standardized to 14 mm in length.

Initial characterization

Initial X-ray was taken and corroborate duct permeability; limes type K #10 were used, radiographic images with conductometry were taken with a manual type instrument K #15. 1 mm was used as actual working length obtained short of the radiographic apex.

Table 1: Comparison of effectivity between radicular sealing

Characteristics	AH Plus® (1)	EndoSequence BC sealer® (2)	MTA Fillapex® (3)	Ref.
Radiopacity	1>2	-	-	9
Fluency	-	2>1	-	9
Sealing capacity (unique cone)	-	1=2, 2 needs more time	-	10
Adhesion without gutta-percha	1>3	-	-	11
Adhesion force	-	2>1,3	1>3	12-14
Dentine penetration	1=2	-	3>2	15,16
Retreatment success	1=2	-	-	15

Moreover, conduits were made by a single operator using nickel titanium rotary instruments TF Adaptive 50.04, 23 mm, a 30-gauge needle was used to irrigate between each instrument. Irrigation was carried out with a solution of 5.25% sodium hypochlorite (NaOCl) and recapped between each instrument with K file #15, NaOCl was activated with ultrasound 3 cycles of 20 s at the end of each instrumentation.

All experimental groups were irrigated with 3 mL of 17 % EDTA, the chelating agent with ultrasound activated with a support for type U files, with a type U #20 file, 3 cycles of 20 s. Each sample received a final irrigation of 5 mL with 5.25% sodium hypochlorite. Ducts with paper points #50 were dried.

Forty-five random samples were divided for 3 groups of 15 each, which were filled with: GROUP 1: BC Sealer single cone; GROUP 2: MTA Fillapex with lateral condensation; GROUP 3: AH Plus lateral condensation and gutta-percha as control group was used.

Obturation

Once prepared the sample ducts, the obturation procedure was performed. Group 1 was sealed using single cone technique and sealer cement BC Sealer, with a cone point™ number 50.04 BC was used, the excess portion of gutta-percha was cut and was vertically

compacted. Group 2 was sealed using the cold lateral condensation technique with standardized gutta-percha and sealer cement MTA Fillapex, once the lateral condensation, excess portion of gutta-percha was removed and vertically compacted. Finally, Group 3 was sealed using the cold lateral condensation technique with standardized gutta-percha and AH PLUS cement sealer, once the lateral condensation was done, the extra portion of gutta-percha was taken off and compact vertically compacted.

After a week, the roots were placed in a cylindrical mold and vertically filled with polyester resin. Samples were stored at room temperature for 24 hours at a temperature of 37°C. Each root was horizontally sectioned at a 4 mm thickness approximately in the middle and apical third using a diamond disc in cooled water.

Adhesion force test

A couple of specimens were obtained by prepared tooth, thus leaving 30 samples in each group. The specimens were taken to be analyzed with the SHIMADZU universal machine for mechanical tests using a metal punch designed with the approximate diameter of gutta-percha, this needle-like device allowed the exertion force on the mass of gutta-percha vertically [17], the punch was placed in a test tube with a 1/8 borehole, at one end the punch was fixed with epoxy resin, once fixed to the upper jaw, in the lower jaw was placed the sample, and measured the area of the gutta-percha. For calibration purposes, the machine was activated to zero and then the compression test was started for each of the samples (Figure 1).

To obtain the results of the force applied to the gutta-percha, the maximum effort to displace the gutta-percha was recorded. Data was collected in files according to endodontic sealer cement type. The adhesion strength was calculated by dividing the maximum breaking load between the duct area of each specimen using the following formula:

$$\sigma = P/V \text{ (Stress=Force/Area)} \quad (1)$$

For megapascals (MPa) unit conversion, data was analyzed with a program that allows finding significant differences between the groups.

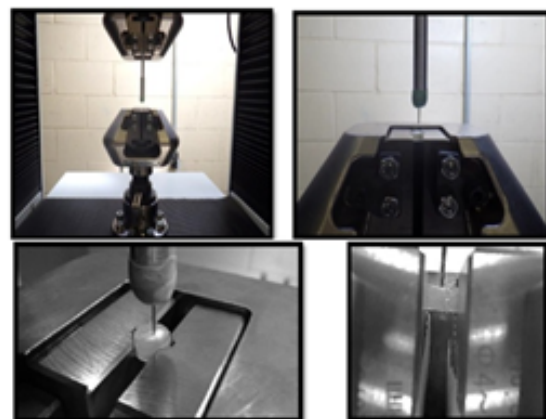


Figure 1: Adhesion force test "push out", using a metallic punch

Scanning electron microscopy (SEM)

Studies of scanning electron microscope were performed in a field emission microscope JEOL JSM 7600F, non-conductive samples; require prior preparation to be analyzed. A little graphite tape was placed in a metal sample holder. A small portion of the test sample was cut and placed on the tape. Then, these samples were coated with a thin layer of a metal assisted deposition at a voltage around 1 kV for 2 minutes.

Thereafter, samples were placed on a baking electron microscope and images were captured in different areas at different magnitudes of amplification.

Statistical analysis

The study was conducted under the statistical program SPSS for Windows version 21 considering a significance level of 0.05 for studying, performing statistical Kruskal Wallis.

RESULTS

A transversal and *in vitro* comparative experimental study was performed, in order to compare the adhesive strength of the sealers, 3 cements with different obturation techniques were carried out. This evaluation was not statistically significant different between groups using the Kruskal Wallis test and the average since these tests are used when data do not follow a normal distribution.

Adhesion force test

Among the most important results it can be mentioned that for Group 1: BC Sealer single cone, the adhesion strength to the middle third had a value of 0.1221 MPa and for the third apical a necessary strength of 2.238 MPa. In the case of Group MTA Fillapex Lateral Condensation, the adhesion strength in the middle third was 0.1156 MPa and the apical third of 2.248 MPa was obtained. Finally, the Group cement sealer AH Plus-Condensing Side, the middle third obtained a value of 0.06538 MPa, with respect to the apical third is 1.091 MPa.

Performing comparison among the three cements, in the middle third, BC Sealer required more force to displace the gutta-percha (P=0.1221 MPa), followed by MTA Fillapex (P=0.1156 MPa) and AH Plus (P=0.0654 MPa). On the other hand, the apical third MTA Fillapex required more force to displace the gutta-percha (P=2.248 MPa), followed by BC Sealer (P=2.238 MPa) and AH Plus (P=1.091 MPa).

Therefore, BC Sealer requires more force to displace the gutta-percha in the middle third, and in the apical third was MTA Fillapex (Figure 2).

Scanning electron microscopy (SEM)

Figure 3.1, report a general aspect of a transversal section of the tooth where can be observed a fracture that begin from the internal diameter to the external surface (yellow arrows). In this figure, it can be seen that the internal surface of the apical hole is plane, hence there is no evidence of mechanic damage of the material, it can be assumed that there is not a chemical interaction. It's

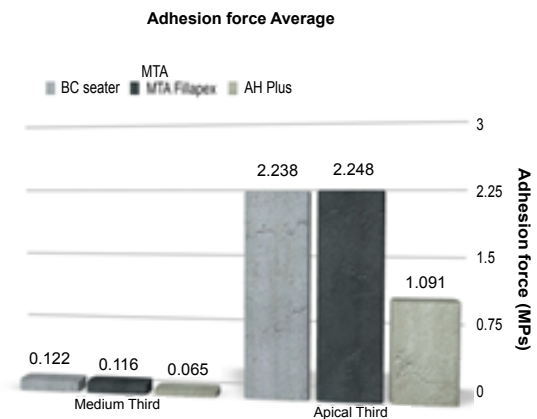


Figure 2: Average of adhesion force of middle and apical third of three cement sealers, where BC Sealer shows a major force to displace the gutta-percha in middle third and for the apical third was MTA Fillapex

important to emphasize that this zone is related with the gutta-percha/adhesive interface (there was no evidence of chemical interaction in this zone).

Figure 3.1 shows a semiquantitative analysis of the further zone of the apical hole (zone 1), where it was identified the main chemical composition of the tooth Ca, P, N, F, Na, Mg and Cl.

Figure 3.2 shows a semiquantitative analysis of the internal zone of the apical hole (zone 2), where was identified as chemical composition Ca, Zr, Si and P, which correspond to the chemical composition of the adhesives. Figure 3.2 shows a closest view of the fracture where it can be observed a plane surface which there is no evidence of mechanic damage of the material, it can be assumed that these fractures are associated to the compression stress force originated from the encapsulating step of the tooth in the acrylic resin.

Figure 4 shows gutta-percha of sample 2A14. Figure 4.1, shows a general aspect of the gutta-percha where can be appreciated adhesive residues with a white aspect (yellow lines), also can be observed as overlapping of the material and fractures. 70% of the gutta-percha surface

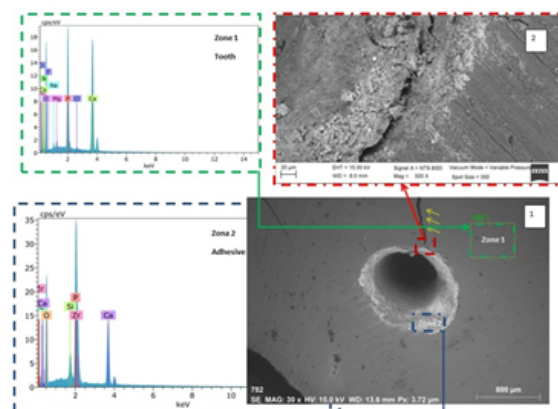


Figure 3.2: Semiquantitative analysis of the internal and further zones of the apical hole

is free of the adhesive; there is no presence of scratches of the surface. With these observations it can be proposed that there is no chemical interaction between the gutta-percha/adhesive interfaces. In Figure 4.2, shows a closest view of the surface morphology of the overlapping zone, where there is no evidence of mechanic damage of the material, hence it can be proposed that there is not chemical interaction between the adhesive and gutta-percha. The spectrum of Figure 4.1 shows a semiquantitative analysis of the adhesive (zone 1), which were identified as main chemical elements Ca, W, Zn, Ti and Al, which correspond to the chemical composition of the adhesive. Figure 4.2 represents the semiquantitative analysis of the gutta-percha zone (zone 2), which were identified the following chemical elements C, Na, Si, Ca, Al, S and Zn, which correspond to the chemical composition of the gutta-percha.

Figure 5 describes the apical hole of sample 2A14, where in Figure 5.1 shows a general aspect of the transversal section of the tooth, which can be observed fracture which grows from the external diameter to the apical hole

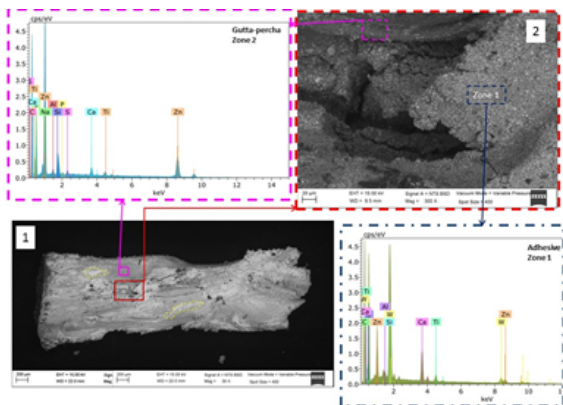


Figure 4: Evaluation of the gutta-percha/adhesive interface of sample 2A14

(yellow concave arrows). In this image it can be seen that the internal surface of the apical hole is plane and there is no evidence of mechanic damage of the material, it can be proposed that there is no chemical interaction. It is important to emphasize that this zone is related with the gutta-percha/adhesive interface (there was no chemical interaction in the interface). Also it can be observed that fractures present a pattern of the concave growth which starts from the external surface to the apical hole (yellow concave arrows). These observations give evidence that the fractures were generated from compression stress forces associated with the encapsulating process of the teeth in the acrylic resin.

Figure 5.2 shows a closest view of the internal surface of the apical hole, which can be observed a plane surface, there is no adhesive residue neither scratching of the zone, there is no evidence of damage, and hence there is no evidence of chemical interaction. Figure 5.3 presents a closest view of the perimetral diameter section which can be seen in two zones, with a white color corresponding to the adhesive (zone 1), and zone 2 without adhesive presence. In this figure, a rough surface associated to a scratching or damage of the surface, demonstrating a

chemical interaction between adhesive and tooth can be seen.

A semiquantitative analysis of the tooth zone (zone 2) shows the following chemical elements Ca, P, Na, Mg, Si and Cl commonly present in tooth, and the chemical composition of the perimetral zone of the apical hole (zone 1) are Ca, P, Si, W, Na, Ti and Cl which are typical of the adhesive chemical composition. According to the present observation it can be proposed that there was only a partial chemical interaction between the tooth/adhesive interfaces, which correspond with the force values reported before.

Figure 6.1 represent a general aspect of the apical hole which can be appreciated adhesive residues and fractures on the tooth surface, also in the middle area

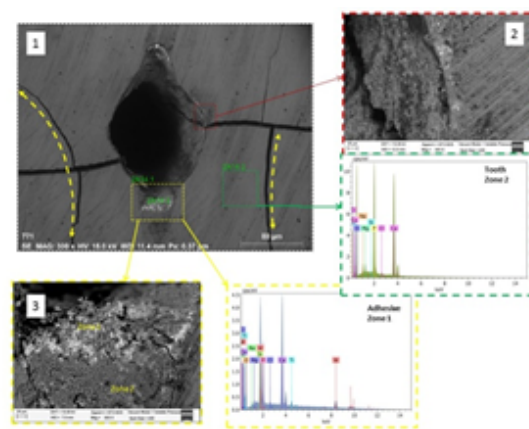


Figure 5: Chemical interaction in analysis of adhesive/tooth interface in the apical hole of sample 2A14

of the apical hole (yellow arrows). Figure 6.2 shows a closest view of the apical hole, where fractures and mechanical damage can be seen. A semiquantitative analysis was evaluated obtaining the following chemical composition: Ca, O, Ni, Al, Si, P and Mg (red field) and Ca, O, Na, Mg, Si and P (purple field). In Figure 6.3 can be observed the propagation of the fracture when the adhesive dissociates from the teeth. Finally, in Figures 6.4 and 6.5 can be seen the fracture zone (adhesive/tooth interface) where a plane surface can be observed. Chemical composition of the adhesive in Figure 6.4 obtaining the following elements Ca, O, Na, Al, Mg, Si and P (orange field) was analyzed. In this figure (red arrows) can be observed zones with release of the adhesive (spectrum 2). According to the present results we consider that only a partial chemical interaction in the tooth/adhesive interface was present, which is a logical observation and can be related to the push out stress force values reported in this work.

Statistical analysis

The study was conducted under the statistical program SPSS for Windows version 21 considering a significance level of 0.05 for the study, performing statistical Kruskal Wallis analysis. With the value of $p=0.000$ which is less than 0.05 in this study, the adhesion between the three

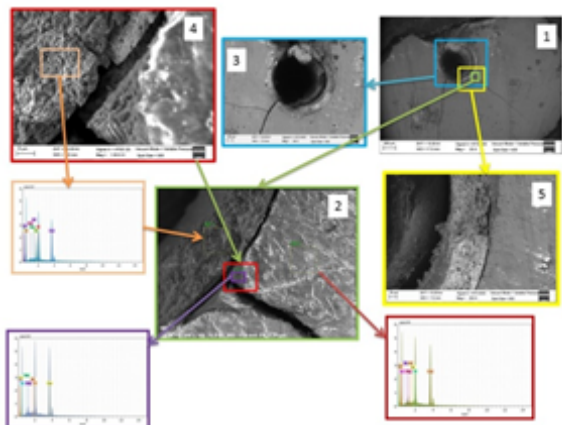


Figure 6: Mechanical damage and chemical analysis of the adhesive/tooth interface

cement sealants BC Sealer CU, AH Plus CL and MTA Fillapex CL are equal.

DISCUSSION

During obturation of root canal, sealers are used to create a bond between the material and the walls of the dentin. Both apical and coronal leakage is a possible cause of failure in endodontics. Therefore, cements should display good properties as adhesive sealers. The bond between the cement and the walls of the duct through the frictional retention or micromechanical adhesion is critical in maintaining the integrity of this interface. Testing adhesion strength is not a fully replicate of the clinical performance of cements and there is no correlation between the binding forces but has proven clinical success, this provides valuable information comparing different sealants cement or sealing techniques.

The expulsion test commonly used to evaluate the bond strength between the duct walls and the cement. Despite being a fulfilled test, used in different studies, it possess a lack of uniformity in the experimental design and the results are often incompatible [11,12].

Christopher DeLong *et al.* evaluated the adhesion forces of MTA Plus (Avalon Biomed Inc, Bradenton, FL), EndoSequence BC-Sealer (BC, Brasseler USA, Savannah, GA) and AH Plus when used in a thermoplastic technique. BC-SC group had the bond strength statistically superior to the MTA Plus-SC and AH Plus-CW groups, so BC-SC and MTA Plus sealant have resistances favorable binding when used in a single cone technique. We agree with Christopher DeLong and colleagues in the results where BC Sealer with a single cone technique was the one with better results for adhesion [12].

Shokouhinejad *et al.* conducted a study to compare the adhesion strength of BC Sealer and AH Plus Sealer in the presence or absence of dentinal sludge. They concluded that, the adhesion strength of BC Sealer was equal to that of AH Plus with or without dentine sludge. In the present study, the dentinal sludge of all groups was removed;

however, we differ with Shokouhinejad *et al.* as BC Sealer with single cone technique performed better than AH Plus to dentin in the absence of dentinal sludge [18].

CONCLUSION

Differences between the groups AH Plus C. L., MTA Fillapex C. L., and BC Sealer C.U., in terms of the mean and median of the adhesion force that was applied in the different thirds of the root canal, BC cement sealer proved the material with better adhesion in both thirds of the root canal, being significantly more noticeable in the middle third. As for the statistical analysis using statistical Kruskal Wallis, it was demonstrated that the adhesion between the three sealers cements BC Sealer CU, MTA Fillapex CL and AH Plus CL are equal in both thirds of the root canal, so there is no significant difference. The three sealer cements are effective for the adhesion in the root canals, any of these can provide an acceptable result when used appropriately. However, according to the results obtained in this investigation, we recommend using BC Sealer, though it requires further study, because is one of the new materials in the market.

CONFLICT OF INTEREST

The authors' declares that they have no conflict of interest.

REFERENCES

1. Michaud RA, Burgess J, Barfield RD, *et al.* Volumetric expansion of gutta-percha in contact with eugenol. *J Endod* 2008; 34:1528-1532.
2. James BL, Brown CE, Legan JJ, *et al.* An in vitro evaluation of the contents of root canals obturated with gutta percha and AH-26 sealer or Resilon and Epiphany sealer. *J Endod* 2007; 33:1359-1363.
3. Paqué F, Sirtes G. Apical sealing ability of Resilon/Epiphany versus gutta-percha/AH Plus: Immediate and 16-months leakage. *Int Endontic J* 2007; 40:722-729.
4. Siqueira Jr JF, Rôças IN, Alves FR, *et al.* Periradicular status related to the quality of coronal restorations and root canal fillings in a Brazilian population. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2005; 100:369-374.
5. Zhang H, Shen Y, Ruse ND, *et al.* Antibacterial activity of endodontic sealers by modified direct contact test against *Enterococcus faecalis*. *J Endod* 2009; 35:1051-1055.
6. Brenes-Dittel G, Moreno-García CM, Ceja AI, *et al.* Comparación de técnicas de sección de dientes tratados endodónticamente para su evaluación al MEB. *Endod Actual* 2012; 7:10-19.
7. Balvedi RP, Versiani MA, Manna FF, *et al.* A comparison of two techniques for the removal of calcium hydroxide from root canals. *Int Endontic J* 2010; 43:763-768.

8. Brito-Júnior M, Leoni GB, Pereira RD, et al. A novel dentin push-out bond strength model that uses micro-computed tomography. *J Endod* 2015; 41:2058-2063.
9. de Miranda Candeiro GT, Correia FC, Duarte MA, et al. Evaluation of radiopacity, pH, release of calcium ions, and flow of a bioceramic root canal sealer. *J Endod* 2012; 38:842-845.
10. Zhang W, Li Z, Peng B. Assessment of a new root canal sealer's apical sealing ability. *Oral Surg Oral Med Oral Pathol Oral Radiol Endod* 2009; 107:e79-e82.
11. Samara Baechtold M, Flávia Mazaro A, Monguilott Crozeta B, et al. Adhesion and formation of tags from MTA Fillapex compared with AH Plus® cement. *Rev Sul-bras Odontol* 2014; 11.
12. DeLong C, He J, Woodmansey KF. The effect of obturation technique on the push-out bond strength of calcium silicate sealers. *J Endod* 2015; 41:385-388.
13. Ozkocak I, Sonat B. Evaluation of effects on the adhesion of various root canal sealers after Er: YAG laser and irrigants are used on the dentin surface. *J Endod* 2015; 41:1331-1336.
14. www.scielo.org.mx/pdf/rmib/v37n2/2395-9126-rmib-37-02-00115.pdf
15. Kim H, Kim E, Lee SJ, et al. Comparisons of the retreatment efficacy of calcium silicate and epoxy resin-based sealers and residual sealer in dentinal tubules. *J Endod* 2015; 41:2025-2030.
16. McMichael GE, Primus CM, Opperman LA. Dentinal tubule penetration of tricalcium silicate sealers. *J Endod* 2016; 42:632-636.
17. González-Vizcarra B, Carrillo-Varguez AG, Villarreal-Gómez LJ, et al. Fabricación de un Punzón para la Realización de Ensayos para Evaluar la Fuerza de Empuje en Selladores Dentales. *Memorias Del XXI Congreso Internacional Anual de la SOMIM Tema A2b Manufactura* 2015.
18. Shokouhinejad N, Gorjestani H, Nasseh AA, et al. Push-out bond strength of gutta-percha with a new bioceramic sealer in the presence or absence of smear layer. *Aust Endod J* 2013; 39:102-106.